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THE ROOM TEMPERATURE MOISTURE KINETICS OF KEVLAR 49 FABRIC/EPOXY LAMINATES

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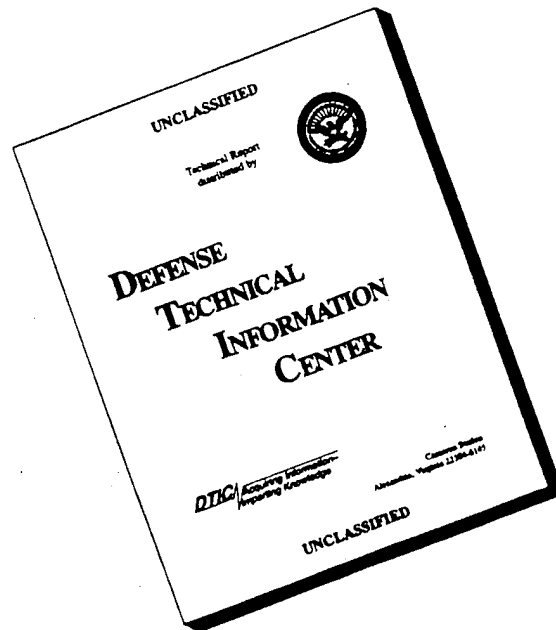
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ABSTRACT

Hygrothermal effects can significantly influence the failure behavior of high performance epoxy-based composites. As such, it is necessary to determine, for one, the moisture diffusion rates in the composite's principal axes so that moisture densities and distributions can be estimated for life-cycle exposures. In this study, moisture diffusion coefficients are determined for quasi-isotropic Kevlar 49 181-style fabric reinforced Narmco 5208 epoxy laminates, and for the epoxy and fabric components individually. A microbalance technique is employed to continuously monitor the percentage weight gain of specimens exposed to 76 percent relative humidity at room temperature. The anisotropy of moisture diffusivity in the composite laminates is determined by use of a slope ratio (absorption vs time) technique and specimens of appropriate geometry. Results show that moisture diffuses in the laminate plane $(6.1 \times 10^{-8} \text{ cm}^2/\text{sec})$ two orders of magnitude faster than through the thickness $(1.7 \times 10^{-10} \text{ cm}^2/\text{sec})$. These values compare to a resin diffusivity of $6.5 \times 10^{-10} \text{ cm}^2/\text{sec}$. It is speculated that the rapid inplane diffusion is due to the preferential diffusion of moisture in the filament along its length.

INTRODUCTION

The hygrothermal response problem of epoxy resins used as composite matricies in aerospace structures is well known and is being extensively investigated [1-11]. These studies show that moisture, when absorbed into the polymer network, results in a dilatation of the resin and in a lowering of the glass transition temperature. Those physical changes can lead to an internal stress buildup or redistribution, to a loss of critical tolerances, or to a lowering of the composite service temperature capabilities. All of these effects will reduce overall composite performance. The majority of studies on environmental effects in composite materials have been conducted on the graphite/epoxy, glass/epoxy and boron/epoxy systems. In those systems, the inorganic filaments do not absorb moisture and the study of environmental sensitivity can be concentrated on the matrix.

Kevlar/epoxy composites can also be applied to aerospace applications which require long term environmental exposures and high service temperatures. The Kevlar/epoxy system is particularly interesting in that the fiber itself has been shown to absorb significant quantities of moisture [12-13]. In addition, the low compressive strength of the filament dictates that a stiff matrix is required to prevent filament buckling at low loads. A lowering of the matrix glass transition temperature below the service temperature will result in a drastic loss in the composite mechanical properties. It is important, therefore, to understand the diffusion behavior of moisture in Kevlar/epoxy laminates in order to predict moisture concentration profiles for lifetime exposures.

This paper presents the results of an investigation of the diffusion kinetics of moisture into Kevlar/epoxy laminates and into the constituent

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where a is an exponent reflecting the order-of-magnitude difference between the two diffusion coefficients. The individual values for D_x and D_{yz} are obtained by combining Eqns. (3) or (4) and (7). Thus, for example, D_x is given by

$$D_x = \left\{ \frac{S_1 h_1 \sqrt{\pi}}{4M_m} \Big/ \left(1 + \frac{2h_1}{\ell_1} 10^{a/2}\right) \right\}^2 \tag{8}$$

while

$$D_{yz} = 10^a \left\{ \frac{S_1 h_1 \sqrt{\pi}}{4M_m} \Big/ \left(1 + \frac{2h_1}{\ell_1} 10^{a/2}\right) \right\}^2 \tag{9}$$

The slope ratio can be solved for in terms of the sample dimensions and the diffusion difference exponent by combining Eqns. (5) and (7). The result is

$$S_R = \left(\frac{h_2}{h_1} + \frac{2h_2}{\ell_1} 10^{a/2}\right) \Big/ \left(1 + \frac{2h_2}{\ell_2} 10^{a/2}\right) \tag{10}$$

This equation reflects the fact that the sensitivity of the slope ratio to the diffusion difference exponent is critically dependent on the choice of dimensions h_1, h_2, ℓ_1, and ℓ_2. This dependence is manifested through ratios of the dimensions.

Finally, there is an additional consideration in regard to these dimensions. Specifically, composite blocks having equal total surface areas are used because of the possibility of moisture adsorption artifacts in the absorption data. This equality of total surface areas requires that sample size selection be based on the relation

$$2\ell_1^2 + 4h_1\ell_1 = 2\ell_2^2 + 4h_2\ell_2 \tag{11}$$

Eqn. (11) can be used to select the magnitude of ℓ_2 once h_1, h_2, and ℓ_1 have been chosen. Typically, h_1, h_2 and ℓ_1 are first chosen through trial and error in the light of the sensitivity considerations of Eqn. (10). An estimate of the expected value of a is required for assessing the latter.

EXPERIMENT

The moisture diffusivity measurements in this study were conducted on a Cahn 100 microbalance adapted such that the specimen chamber could be exposed to a desired environment. This method offered the advantages of a high degree of accuracy (± 0.05 mg) and of continuous monitoring of the specimen, eliminating the possibility of errors due to specimen removal for weighing. These features enabled an accurate account of weight gain versus time to be determined in a few days so that many specimens could be exposed in a short period of time.

The composites examined in this study were taken from a [0, 90, ±45], 6.35 mm thick plate of Dupont Kevlar 49, 181 style fabric in a Narmco 5208 epoxy matrix. The composite was autoclave molded from preimpregnated sheets to a final cure temperature of 175°C and then post cured in an air circulating oven for 4 hours at 190°C. Moisture exposure specimens were machined to size by diamond grinding. Neat resin specimens were ground from a 5208 casting supplied by Narmco Materials, Inc.

Specimen sizes were chosen such that they would fit into the 20.3 mm diameter chamber and would be of equal surface area to eliminate possible distortions in slope ratio by adsorption effects. The resultant specimen dimensions were 5.08 mm x 15.80 mm x 15.80 mm and 1.27 mm x 19.00 mm x 19.00 mm calculated from Eqns. (10) and (11). Two composite specimens of each size and three resin specimens, two of the 5.08 mm thick and one of

phases. The effect of composite anisotropy on moisture diffusivity is examined with an experimental method based upon the ratio of weight gain versus time slopes for specimens of different thickness.

THEORY

The short-time moisture weight gain of laminated composite blocks having edge-lengths of comparable dimension has been described by Shen and Springer [14] as

$$M = \frac{4M_m}{h\sqrt{\pi}} \left(\sqrt{D_x} + \frac{h}{\ell}\sqrt{D_y} + \frac{h}{n}\sqrt{D_z} \right)\sqrt{t} \quad . \tag{1}$$

In this equation, M represents the instantaneous weight percent of moisture gain, i.e.

$$M = \frac{\text{Wet Weight} - \text{Dry Weight}}{\text{Wet Weight}} \times 100 \quad . \tag{2}$$

The factor M_m above represents the maximum long-term equilibrium moisture content. The parameters h, ℓ, and n designate, for example, thickness in the x-direction, width in the y-direction, and length in the z-direction, respectively. The quantities D_x, D_y, and D_z denote the associated moisture diffusivities, i.e., moisture diffusivities along the coordinate directions. Finally, t denotes time.

For the composite layups that give equal in-plane diffusion coefficients, D_x, and $D_{yz} = D_y = D_z$ through separate diffusion experiments on samples of two different sizes. Specifically, if one considers samples with dimensions h_1, ℓ_1, and $n_1 = \ell_1$ for the first tests, then Eqn. (1) gives

$$\left(\frac{M}{\sqrt{t}}\right)_1 = \frac{4M_m}{h_1\sqrt{\ }} \left(\sqrt{D_x} + \frac{2h_1}{\ell_1}\sqrt{D_{yz}} \right) = S_1 \quad , \tag{3}$$

where S_1 represents the initial slope of a plot of M versus $\sqrt{t}$. For a second set of tests on samples having dimensions h_2, ℓ_2, and $n_2 = \ell_2$, we have similarly

$$\left(\frac{M}{\sqrt{t}}\right)_2 = \frac{4M_m}{h_2\sqrt{\ }} \left(\sqrt{D_x} + \frac{2h_2}{\ell_2}\sqrt{D_{yz}} \right) = S_2 \quad . \tag{4}$$

A division of Eqn. (3) by (4) gives after cancellations

$$\frac{D_{yz}}{D_x} = \frac{1}{4} \left\{ \frac{\frac{h_2}{h_1} - S_R}{\frac{h_2 S_R}{\ell_2} - \frac{h_2}{\ell_1}} \right\}^2 \quad , \tag{5}$$

where

$$S_R = \frac{S_1}{S_2} \tag{6}$$

denotes a slope ratio calculated from the slopes recorded in the two experiment sets. Eqn. (5) thus gives a theoretical basis for determining the relative magnitudes of the diffusion coefficients D_x and D_{yz} from the slope ratio and sample dimensions. This result can always be written as

$$\frac{D_{yz}}{D_x} = 10^a \quad , \tag{7}$$

where a is an exponent reflecting the order-of-magnitude difference between the two diffusion coefficients. The individual values for D_x and D_{yz} are obtained by combining Eqns. (3) or (4) and (7). Thus, for example, D_x is given by

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the chamber, the composite specimens were redried in vacuum and the fabric volume fraction determined by chemical dissolution [15]. A summary of the microbalance absorption results is given in Table 1.

The equilibrium moisture content of the composite and its constituents as a function of relative humidity was determined by placing thin specimens of each in controlled humidity atmospheres and monitoring the specimen weight gain until equilibrium was reached. The specimen weights were then periodically monitored for 2500 hours. The composite specimens were 25 mm squares cut from a single ply fabricated with the same procedures as the 6.35 mm thick plate. The resultant composite was determined to have a fabric volume fraction of 49 percent. Neat resin specimens 0.5 mm x 10.0 mm x 25.0 mm were ground from the Narmco casting for the equilibrium exposures. Twenty-five mm squares of 181 Kevlar 49 were used for determining the equilibrium fiber moisture content. The fabric specimens were taken from the composite prepreg from which the resin was removed by dissolution in acetone. The equilibrium moisture content versus relative humidity results are plotted in Fig. 2. Least squares fits to the data are also plotted in Fig. 2.

DISCUSSION

A knowledge of the material equilibrium moisture content is necessary prior to a moisture diffusivity calculation from Eqn. (1). As seen in Fig. 2, the Kevlar 49 fabric and composite equilibrium moisture content data appear to be a straight line function of relative humidity. The 5208 resin data shows more deviation from the least squares fit, but still has a coefficient of determination of 0.99. Such agreement is considered acceptable for the purposes of this discussion. It should be noted that other investigators [6] have found equilibrium data for 5208 to be better fit by a power law rather

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the 1.27 mm thick, were examined. In addition, a 19 mm square of 181 style Kevlar 49 fabric was examined. A 0.25 mm diameter hole was drilled in the solid samples to allow attachment of the microbalance suspension wire,

Prior to exposure in the microbalance, all specimens were cleaned with ethanol and baked overnight at 120°C in vacuum to remove any post-fabrication moisture pickup. The specimens were then cooled under vacuum, weighed on an analytical balance and stored in a desiccator. Specimen weights after storage agreed with the as-dried weights, which indicated that no moisture was lost to the desiccant. After a specimen was weighed, it was attached to the microbalance suspension wire, and the chamber was evacuated to less than 30 mtorr for approximately one hour. Again, no weight change was evident. The chamber was then back filled with dry air to 620 torr (ambient). A 76% relative humidity environment maintained over a saturated NaCl solution in an adjacent chamber was then valved into the microbalance chamber, and the specimen weight gain versus time was recorded continuously on a strip chart recorder. All microbalance measurements were conducted at a nominal ambient temperature of 21°C.

The initial appearance of a representative absorption curve is shown in Fig. 1. After a slope of weight gain versus square root of time could be established, the salt solution chamber was valved off from the specimen, and the specimen chamber was evacuated. The resultant desorption slope was equivalent to the absorption slope for each sample. In addition, the desorption curves remained linear, and passed through zero. This desorption behavior indicates that the short-time (<24 hours) response of the absorption curves is due to a lack of moisture equilibrium in the test chamber. As seen in Fig. 1, approximately a day is required to reach an equilibrium boundary condition at the specimen surface. Upon removal from

To apply the slope ratio technique to the composite data given in Table 1, average values of thickness, length and slope are determined for each specimen size. The resultant slope ratio is 1.0705. Eqn. (5) is solved with the average dimensional values and reveals that D_{yz} (composite) = 354 D_x (composite), i.e., moisture diffuses in the plane more than two orders of magnitude faster than through the thickness of these Kevlar/epoxy laminates.

D_x and D_{yz} of the composite can be determined by solving Eqns. (9) and (10). This results in values of D_x (composite) = 1.7×10^{-10} cm^2/sec and D_{yz} (composite) = 6.1×10^{-8} cm^2/sec. In comparison to the 5208 diffusivity, D_x is 3.7 times slower but D_{yz} is nearly 100 times faster. Such figures would indicate that either the Kevlar 49 fibers or the Kevlar/epoxy interface is providing a preferential diffusion path to moisture in the laminate plane.

To calculate the diffusivity of the Kevlar fabric, it is necessary to assume a model. The assumptions for two simple diffusion models are (1) that all diffusion takes place radially through the filament, thereby ignoring end effects, or (2) that all diffusion occurs along the filament axis and thereby ignoring surface effects. Clearly, a combination of the two would more nearly mirror reality; however, with the limited data available in this study, these approaches can be used to bound the problem.

To solve case 1, the fabric is modeled as consisting of 2 yarns/mm, each composed of 267 square fibrils 0.0316 mm on a side. Substituting that value for h into Eqn. (1) and solving for four sided diffusion yields a value of D_{Kevlar} (= D_x = D_y) = 4.2×10^{-13} cm^2/sec. This value can be considered an upper bound for diffusion normal to the filament in that the filaments are modeled to act independently with no filament-filament contact. To solve Eqn. (1) for case 2, h is assumed to be the 19 mm specimen length and again four sided diffusion is assumed. Case 2 results in D_{Kevlar} = 9.8×10^{-7}

than a least squares function. The composite data in Fig. 2 closely follows a rule of mixtures relationship to the filament and matrix until high humidities. At high humidities, the composite appears to be fiber dominated. The reasons for this are unclear, however, the resin behavior appears more nonlinear at high humidity. Such behavior could be due to a difference in degree of cure, void content, residual curing stresses, etc. between the neat resin casting and the composite matrix. The equilibrium moisture contents of the epoxy, fiber and composite taken from Fig. 2 at 76 percent relative humidity are 4.8, 3.75, and 4.0 percent respectively.

The sensitivity of the microbalance technique employed in this study can be determined by examining the variability in the measured diffusivity of the three resin samples. By assuming the resin to be isotropic ($D_{resin} = D_x = D_y = D_z$) and solving Eqn. (1), the moisture diffusion coefficients determined for the 5208 epoxy specimens are 7.1×10^{-10} cm^2/sec, 6.6×10^{-10} cm^2/sec and 5.8×10^{-10} cm^2/sec. These values result in an average $D_{5208} = 6.5 \times 10^{-10}$ cm^2/sec. with a coefficient of variation of 10.3 percent.

The effect of experimental variability on the slope ratio technique can be seen by calculating $(D_{yz}/D_x)^{1/2}$ for the different size epoxy specimens. Since the resin specimens are assumed to be isotropic, D_{yz} should be equal to D_x. Substituting the values of thickness, length and slope ratio (average values of the 5 mm thick specimens) for the specimens into Eqn. (5) results in a value of D_{yz} = .59 D_x for a slope ratio of 2.935. It can be seen that experimental errors are compounded through calculations of this type and that small differences in determined diffusivity on the order of those calculated for the resin above are not significant. However, large differences in D_x and D_{yz} determined by the slope ratio technique should be valid.

cm²/. sec. This value (Case 2) is of the order of magnitude one would expect for the Kevlar filaments to explain the in-plane diffusion rate of the composite.

While these values are not presented as hard evidence, one may speculate that the rapid diffusivity of moisture in the laminate plane is due to a wicking action ($D_{axial} >> D_{radial}$) along the Kevlar filament axis. To further test this hypothesis, an additional composite specimen was fabricated with a much lower filament volume fraction. Processing conditions were maintained identical to those of the previously tested specimens. The resultant specimen dimensions and moisture absorption slope is also listed in Table 1.

In order to compare the lower volume fraction specimen with the previous specimens, it is necessary to calculate a bulk diffusivity, D. Eqn. (1) can be rewritten as [14]

$$M = \frac{4M_m}{h\sqrt{\pi}} \sqrt{D} \sqrt{t}$$ (12)

where

$$D = D_x\left(1 + \frac{h}{\ell}\sqrt{\frac{D_y}{D_x}} + \frac{h}{m}\sqrt{\frac{D_z}{D_x}}\right)^2$$ (13)

A value of D = 1.5 x 10⁻⁹ cm²/sec can be calculated for the low volume fraction composite from Eqn. (12). This compares to a D of 3.4 x 10⁻⁸ cm²/sec for a comparable higher volume fraction specimen. Thus, a 31 percent reduction in fabric content reduces the bulk diffusivity by 22 times. It is felt that this data further indicates that Kevlar filaments are highly anisotropic to moisture diffusion and that the dominant mode of moisture absorption of Kevlar filaments and their derived composites is along the filament axis.

Such a mechanism is also consistent with the microstructure of highly aligned fibrils with no chain molecules crossing and supporting axial planes, and having only weak intermolecular bonds between planes, as has been seen in tensile and fatigue tests of Kevlar 49 filaments [16].

CONCLUSIONS

The results of this study on the diffusivity of moisture in Kevlar 49/epoxy laminates led to the following conclusions:

(1) The equilibrium moisture content, M_m, of a Kevlar/epoxy composite is an average of the filament M_m and matrix M_m at low humidities but may be filament dominated at high humidity levels.

(2) The diffusion coefficient of moisture in 5208 resin is 6.5 x 10⁻¹⁰ cm²/sec with a coefficient of variation of 10 percent.

(3) The diffusion coefficients of a quasi-isotropic 52 v/o Kevlar/epoxy 5208 laminate are D_x (normal) = 1.7 x 10⁻¹⁰ cm²/sec and D_{yz} (in-plane) = 6.1 x 10⁻⁸ cm²/sec, each with a variation of 10 percent.

(4) A one-third reduction in filament volume fraction results in a twenty-two fold reduction in laminate bulk diffusion.

(5) The rapid in-plane laminate moisture diffusion and its subsequent reduction with filament volume fraction appear to be the result of a highly anisotropic filament diffusivity ($D_{axial} >> D_{radial}$) that preferentially absorbs moisture along its length.

ACKNOWLEDGEMENT

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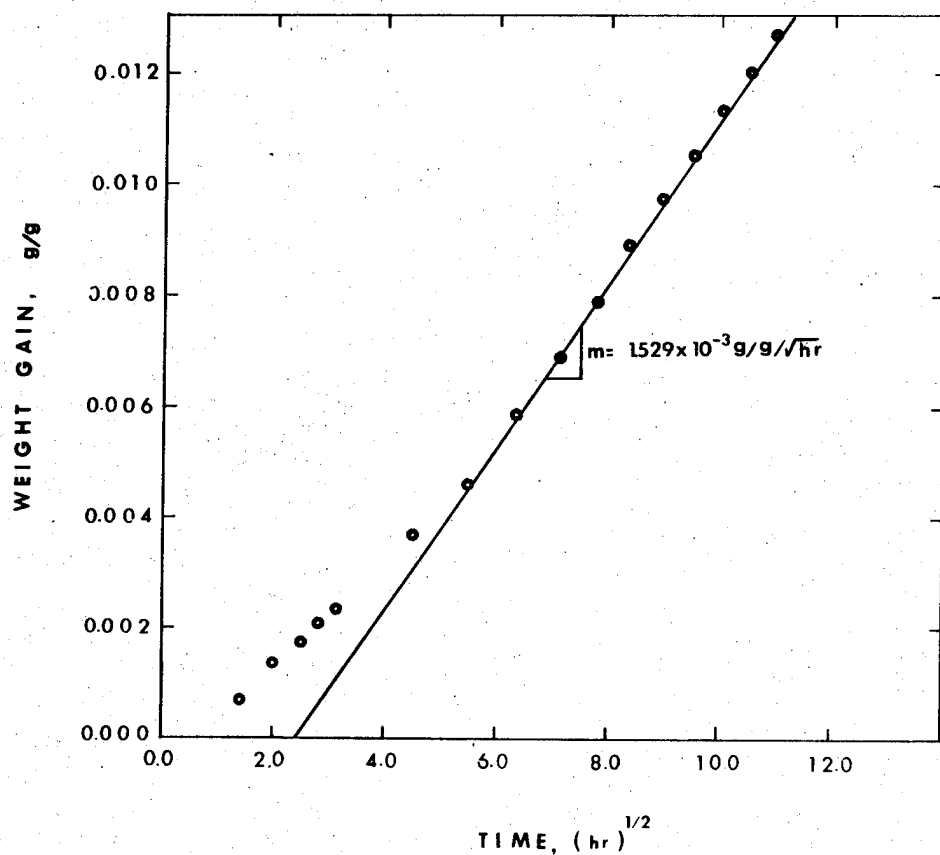(16) Konopasek, L. and Hearle, J. W. S., Journal of Applied Polymer Science, Vol. 21, October 1977, pp. 2791-2815.

Figure 1. Initial microbalance absorption curve for 1.27 mm thick neat epoxy resin (5208) sample.

Table 1

Summary of Microbalance Moisture Absorption Results

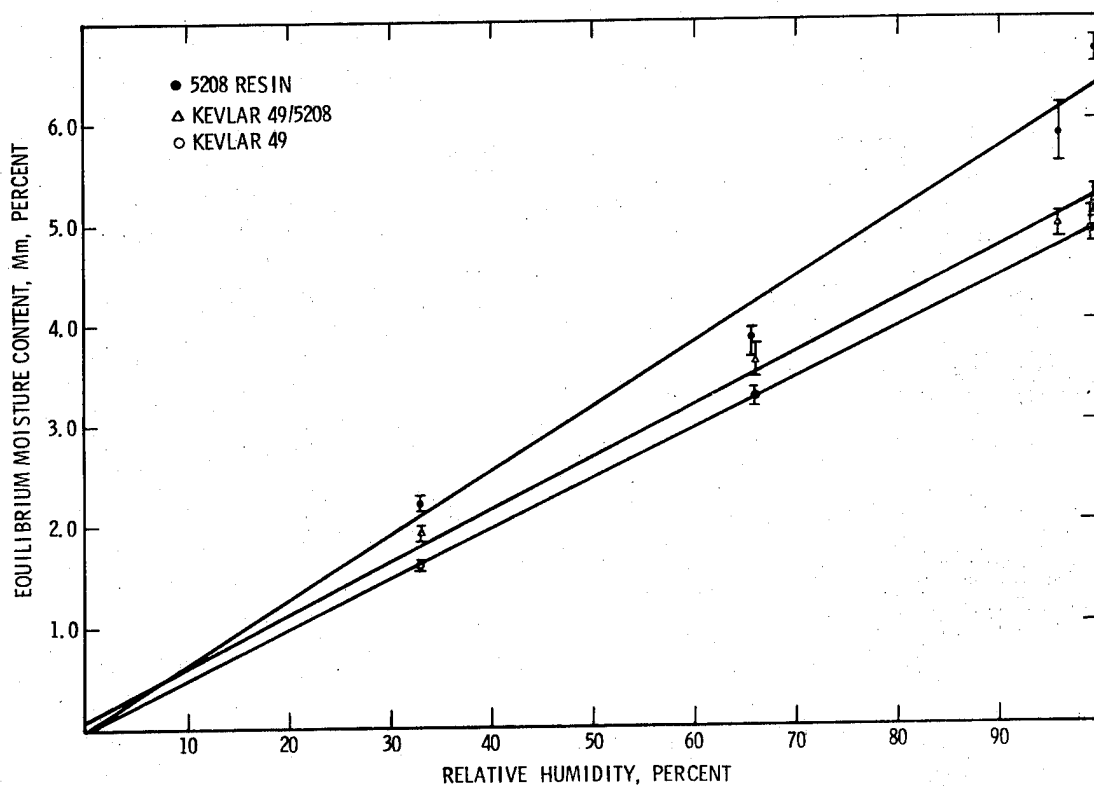| Specimen Description	Thickness mm	Length (width) mm	Slope g/g/√hr
5208 resin	1.28	19.08	.001529
5208 resin	5.12	15.86	.000537
5208 resin	5.07	15.81	.000505
181 Style Kevlar 49 fabric	.28	25.0	.005300
Kevlar 181/5208 (V_f = .52)	1.28	19.04	.002034
Kevlar 181/5208 (V_f = .52)	1.27	19.03	.002186
Kevlar 181/5208 (V_f = .52)	5.08	15.80	.001988
Kevlar 181/5208 (V_f = .52)	5.08	15.81	.001954
Kevlar 181/5208 (V_f = .36)	5.13	15.79	.000450

Figure 2. Maximum moisture content of Kevlar 49 181 fabric/5208 epoxy composite and the constituent phases as a function of relative humidity.